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Cyclic Fatigue Behavior of an Alumina Ceramic with Crack-Resistance Characteristics

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The behavior under cyclic tension–tension loading of an alumina ceramic with pronounced crack-bridging (R-curve) characteristics is studied. Tests on disk specimens with indentation cracks reveal no failures below the static fatigue limit. Theoretical predictions of the stress–lifetime response, based on the premise that environmentally assisted slow crack growth is the sole factor determining lifetime, are consistent (within experimental scatter) with the data. The results indicate that there is no significant cyclic degradation from potential damage to the bridges, at least in the short-crack region pertinent to strength properties. [Key words: fatigue, crack growth, alumina, R-curve.]

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It is well-known that the mechanical properties of metals and polymers are susceptible to degradation under repeated loading, i.e., "cyclic fatigue." Ceramics have generally been perceived as immune to such damage on account of their lack of crack-tip plasticity. Literature studies of cyclic fatigue in ceramics are sparse and inconclusive. Some early work on alumina[1–5] report reduced lifetimes in cyclic relative to static loading. However, analysis of some of these data (specifically data in Refs. 1 and 4) suggests that, within the range of experimental scatter, this reduction can be accounted for in large part by the integrated effect of environmentally enhanced slow crack growth.[6,7] Subsequently, cyclic fatigue studies have been reported on silicon nitride[8–10] and silicon carbide.[11] Again, these studies demonstrate no definitive evidence of true cyclic damage. Indeed, Matsuo *et al.*[8] imply that the fatigue failure times for their silicon nitride are in accord with slow crack growth alone.

Most recently, cyclic fatigue crack propagation has been unequivocally demonstrated,[12,13] in a transformation-toughened magnesia–zirconia ceramic (grain size of 50 μm), using tension–tension loading of compact-tension specimens with "long" cracks (i.e., millimeter-scale, large compared with the microstructure). That work demonstrated crack growth in cyclic loading at stress intensities significantly below those required to generate environmentally assisted crack growth in sustained constant loading, with the extent of the shift in the attendant crack velocity curves depending on the degree of aging of the material. The fatigue effect in that material was attributed, at least in part, to crack-resistance (R-curve) behavior due to shielding by phase transformation, although the detailed micromechanisms remain obscure.

However, it is now established that a wide range of nontransforming ceramics such as aluminas can also exhibit R-curve characteristics, albeit via a different shielding mechanism. In these materials the predominant mechanism of R-curve behavior is persistent grain-localized bridging at the interface behind the crack tip.[14–21] Key to the efficacy of the bridges as toughening agents is the presence of internal thermal expansion mismatch stresses which "clamp" interlocking grains into the alumina "matrix" on either side of the crack interface behind the advancing crack tip. The R curve confers the quality of "flaw tolerance"[22] and, because of its stabilizing influence on crack growth, strongly enhances the "static fatigue" limit.[23] However, there is no direct information on what effect *repeated* loading and unloading might have on the bridging mechanism. In this context we may note that aluminas with more pronounced R curves show a demonstrably greater susceptibility to wear in repetitive abrasion; this increased susceptibility is attributable to cumulative augmentation of tensile internal stresses from the contact damage processes, leading to grain-boundary microfracture.[24] On these grounds we might reasonably expect analogous cumulative damage to the bridges in the repeated loading of propagating cracks, leading to a degradation in the toughness and strength properties. By reducing the crack-tip shielding, such degradation could give rise to cyclic fatigue.

Related effects on the fracture properties of alumina have been described in compression tests on specimens with large notches.[25] There, "fatigue" cracks were observed to initiate from the notches much more effectively in repeated than in sustained loading. As in the interpretation of the wear results referred to above, the enhanced initiation is attributed to grain-boundary microcracking, but driven now by notch-concentrated tensile stresses associated with irreversibilities in the compression stress–strain cycle.[26]

Our objective in the present study was to investigate the effect of cyclic loading on the behavior of a coarse-grained alumina ceramic that shows particularly strong R-curve behavior, specifically in the short-crack region relevant to strength properties. We present results from tension–tension cyclic loading of specimens with controlled indentation flaws in biaxial flexure in water. Extensive indentation-flaw studies on the selected alumina have been previously documented: in inert-strength tests to determine the R-curve parameters[21] and in constant-stressing-rate tests in water to determine crack velocity parameters.[23] We use these "calibrated" parameters in a computer algorithm, tacitly assuming total reversibility of the bridging constitutive law during the loading–unloading cycle, to obtain a lifetime prediction for comparison with the data. The existence of a true cyclic enhancement of fatigue should then be apparent as a systematic tendency for the experimental data to fall below the predicted stress–lifetime curve.

Experimental Procedure

The material used in this study was a commercial polycrystalline alumina ceramic, nominally pure (<0.1% additive) with a mean grain size of 23 μm.[‡] The specimens were in the form of disks, approximately 22 mm in diameter and 2 mm thick.

Controlled Vickers indentation flaws at a load 30 N were placed at the centers of the prospective tensile faces of each specimen. The specimens were mounted into a biaxial loading fixture, with a flat circular punch of diameter 4 mm on a three-point support of diameter 19 mm. The tensile stresses in the loaded specimen surfaces were computed from thin-plate formulas. This is identical to the indentation–strength configuration used previously[23] to obtain lifetime data at *static* applied stresses, which we will adopt here as a convenient baseline for comparing cyclic loading data.

The cyclic load tests were conducted in sinusoidal tension–tension on a servo-hydraulic fatigue testing machine.[§] The minimum tensile stress in each series of tests was maintained at 20 MPa, but the maximum stress was adjusted to coincide with the constant stress levels applied in the static loading tests (covering a practical range of 1 to 10^6 s in lifetime[23]). At least five specimens were tested at each selected peak stress, at frequencies of 1 and 50 Hz, in water. The time/cycles to failure were recorded in each case. In all tests the broken specimens were examined to verify that the failure initiated at the indentation site; exceptions were excluded from the data pool.

Results

Consider first the static fatigue test results in Fig. 1, from Lathabai and Lawn.[23] The solid curve represents an a priori prediction of the static fatigue response, obtained numerically using a computer algorithm[23] based on a combined R–curve and crack-velocity-curve analysis (Appendix): the parameters used in this analysis were "calibrated" from the inert strength and constant stressing rate data for our alumina in previous experiments.[23] The data points are individual experimental breaks; left arrows designate specimens that broke during the loading ramp, right arrows designate long-term "survivors." It is seen that the data scatter uniformly about the theoretical curve, which coincides with the fatigue limit (130±10 MPa) over most of the practical stress range. Any additional fatigue effect in cyclic loading should therefore be manifest as failure stresses *below* this static limit.

The results for individual breaks in cyclic loading tests are plotted in Fig. 2 for the two experimental frequencies used. Arrows at right again designate long-term survivors. The data show a strong similarity in form and spread to those of Fig. 1. The solid curve in Fig. 2 is the integrated theoretical prediction for sinusoidal cyclic loading from the computer algorithm, presuming slow crack growth to be the *sole* source of fatigue and bridge displacements during the unloading to be reversible (Appendix). The level of agreement between theoretical prediction and experimental data is comparable with that in Fig. 1. In accordance with the assumptions above, the effect of cycling is simply to translate the fatigue curve to longer lifetimes, as seen by comparing the solid (cyclic) curve with the dashed (static) curve. The data in Fig. 2 do not fall below the static fatigue limit, indicating that any subsidiary mechanical damage mechanism, if present, plays an insignificant role in the degradation process.

Further indication as to a fatigue effect can be obtained by investigating the role of frequency. It is difficult to see any distinction between 1 and 50 Hz data points, within the scatter, in Fig. 2. Accordingly, we replot the data on cumulative probability diagrams in Fig. 3. The stress range in Fig. 2 is narrowly confined about the fatigue limit, so we include all data in the probability plots so as to increase the statistical sample. Whereas there seems to be a significant shift in the number of *cycles* to failure, no such shift is apparent in the *time* to failure. This is further support for the exclusive role of slow crack growth in the fatigue response.

Discussion

The preliminary results above are confined to one alumina material, and to "short" initial cracks. Nevertheless, the study suggests that we may draw a rather strong general conclusion: that, contrary to the concerns expressed earlier, the micromechanisms responsible for the R curve in nontransforming ceramics *are not necessarily deleterious to the cyclic fatigue response*. We shall return to this important conclusion below.

To account *quantitatively* for the null effect of cyclic loading on the integrity of the bridges for our indentation flaws, it is necessary to resort once more to numerical algorithm. The algorithm contains complete (stepwise) information on the crack evolution for indentation flaws in our alumina–water system. Accordingly, we

‡Vistal grade Al_2O_3, Coors Ceramics Co., Golden, CO.

§Instron Dynamic Testing Machine 1350, Instron Co., Canton, MA.

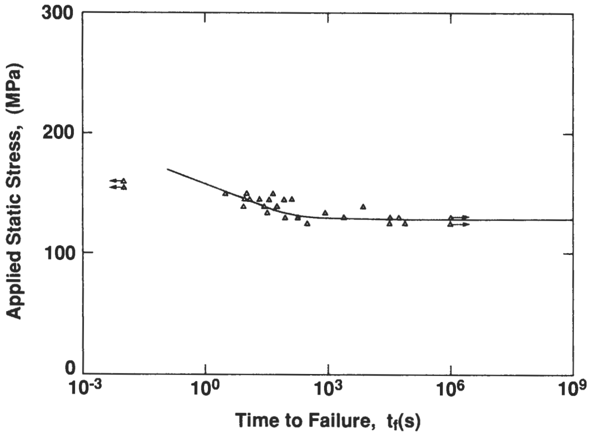

Fig. 1. Static fatigue plot for polycrystalline alumina in water, for Vickers indentations at $P=30$ N. Data points are results of individual tests. Arrows at right designate interrupted tests; at left breakages during ramp loading to maximum applied stress. Solid curve is theoretical prediction. Data from Lathabai and Lawn.[23]

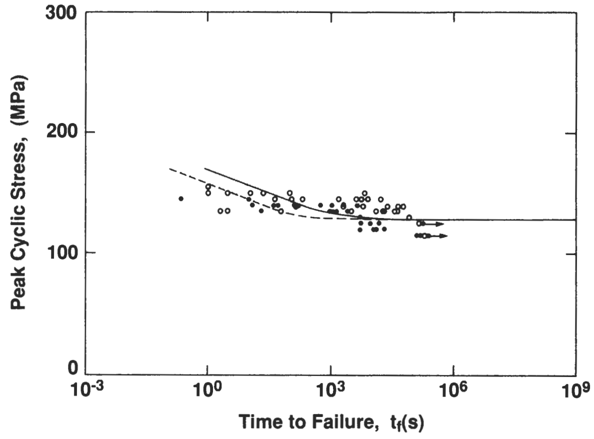

Fig. 2. Cyclic fatigue plot for polycrystalline alumina in water, for Vickers indentations at $P=30$ N. Data points are results of individual tests: open symbols are for 1 Hz, closed symbols, 50 Hz. Solid curve is prediction assuming only slow crack growth. Static fatigue curve from Fig. 1 included for comparison.

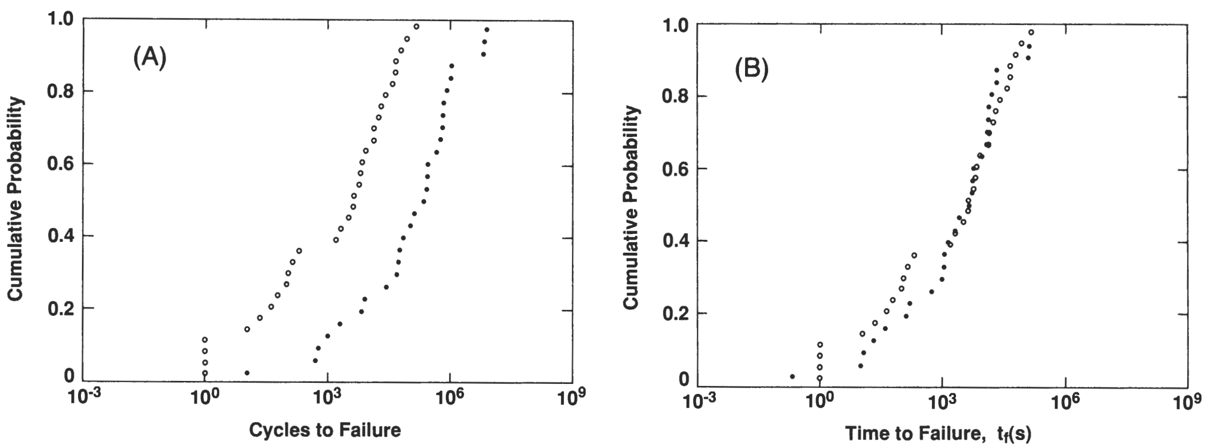

Fig. 3. Cumulative probability plots for (A) cycles and (B) time to failure for data in Fig. 2. Open symbols are for 1 Hz, closed symbols, 50 Hz.

evaluate $2u_Z=0.66$ μm for the crack-opening displacement at the first-intersected bridge, Fig. 4, at failure (corresponding to an instability point part way up the R curve) for *static* applied stresses close to the fatigue limit. This displacement may be compared with $2u_M=2.76$ μm for complete disengagement of the interlocking bridging grains (which would be obtained if the crack were allowed to reach the upper plateau of the R curve), from previous inert strength data.[23] Thus, the ratio $u_Z/u_M=0.24$ is a measure of the critical "pullout strain" for the bridging grains for our indentation cracks. It seems reasonable to suppose that, for ordinary flaws, the grains are in little danger of being effectively "dislocated" from the matrix at this strain.

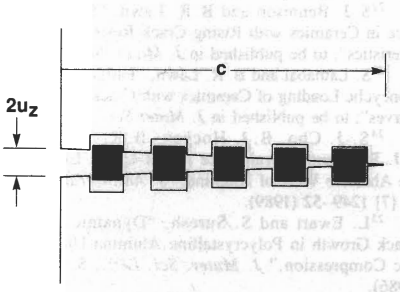

Fig. 4. Schematic showing crack-opening displacement u_Z at edge of bridging zone. At $u_Z=u_M$, grains disengage from matrix.

This estimate is for static loading. In *repeated* loading one might expect irreversibility in the grain–matrix frictional sliding characteristics to contribute adversely to the fatigue response, by permanently degrading the bridges. We envisage two ways this degradation could occur: first, by reducing the frictional resistance at the grain–matrix interface; second, by damaging the surface of the interlocking grains, thereby enhancing transgranular fracture. The indication from our experimental results is that this kind of degradation does not occur to any significant extent, at least for indentation flaws.

In the present study we have considered only *short* cracks. Should we expect the same null behavior for *long* cracks? The mechanisms referred to in the preceding paragraph will surely have a greater effect as the cracks grow farther up the R curve. In extreme cases, beyond the bounds of the R-curve plateau, the interlocking grains will disengage from the matrix ($u_Z>u_M$), such that the bridging zone translates with the crack.[19] There is then additional potential for damage, e.g., from the wedging action of dislodged grains and associated debris at the closed interface. There is some evidence for deleterious fatigue effects in a recent study of long-crack ($\approx$10 mm) specimens of alumina (10-μm grain size),[26] although the authors in that study did not consider the R-curve bridging mechanism and made no attempt at a direct identification of the underlying fatigue mechanisms. The link between short and long cracks in the analysis of fatigue is an important area for further study. Our results would indicate dangers in extrapolating from one crack-size region to the other, e.g., in using fatigue data from long-crack specimens to predict strength behavior.

The present results have important implications. Ceramics processors might seek to tailor microstructures for optimum R-curve properties.[21] However, there is always the concern that such optimization at one extreme of crack size could lead to deleterious behavior at the other. We mentioned the inverse relation between long-crack toughness and wear properties earlier. The present results suggest that similar grounds for concern with regard to a potential correlation between R-curve and cyclic-loading strength properties could prove to be unwarranted.

Appendix

The algorithm used to compute lifetimes for alumina with R-curve characteristics combines the following fracture mechanics elements:[19,22,23]

(i) Fracture mechanics relations and R curve: First, a relation is written for the net driving force on a crack in a material with crack–interface bridging. The stress-intensity factor K_* at the tip of crack length c is given by

$$K_*(c)=K_a(c)+K_r(c)+K_\mu(c) \qquad \text{(A-1)}$$

where $K_a(c)=\psi\sigma_a c^{1/2}$ relates to the applied stress (ψ a geometry constant), $K_r=\chi P/c^{3/2}$ to the contact residual stress at indentation load P (χ a contact constant), and K_μ to the bridging. All the parameters except those in K_μ, which determines the shape of the R curve, are known from indentation studies.

(ii) Constitutive relation: To compute K_μ, one needs the underlying relation between the bridging stress p and crack-opening displacement $2u$ for grain–matrix pullout,

$$p(u)=p_m(1-u/u_M) \qquad \text{(A-2)}$$

where p_m, maximum pullout stress at $u=0$, and u_M, (half) pullout displacement at $p=0$, are expressible in terms of such microstructure parameters as grain size and shape, internal thermal expansion stresses, grain–matrix friction, bridge rupture strain, etc.[22] All these microstructure parameters have been determined for our alumina from inert strength data.[23] In the approximation of "weak toughening," the bridging stress intensity factor is determined from[19]

$$K_\mu(c)=[E/K_*(c)]\int_0^{u_Z(c)} p(u)\,du \qquad \text{(A-3)}$$

where E is Young's modulus. The upper limit, $u_Z(c)$, is (half) the displacement at the edge of the bridging zone, calculable from an expression for the crack profile.[19] This formula is used in conjunction with Eq. (A-1) for both loading and unloading half-cycles, implying full reversibility in the bridge-sliding response.

(iii) Crack velocity relation: The velocity function used is a hyperbolic sine,[23] based on the underlying notion of stress-enhanced thermal activation over atomically localized energy barriers.

$$v(G_*)=v_0 \sinh[(G_*-2\gamma_1)/2\Gamma] \qquad (2\gamma_1\leq G_*\leq 2\gamma_0) \qquad \text{(A-4a)}$$

$$v(G_*)=0 \qquad (2\gamma_1>G_*) \qquad \text{(A-4b)}$$

where γ_0 and γ_1 are surface energies in vacuum and reactive environment, respectively, and v_0 and Γ are adjustable parameters. Note the provision in Eq. (A-4) for a threshold, consistent with the existence of a natural static fatigue limit.[23] To connect Eqs. (A-1) and (A-4), we use the familiar relation $G_*=K_*^2/E$. The adjustable parameters in Eq. (A-4) have been evaluated from constant stressing rate data.[23]

(iv) Fatigue and lifetime relations: The time to failure can be computed numerically for any time-dependent applied stress function, $\sigma_a=\sigma_a(t)$.

$$\sigma_a=const \qquad \text{(static)} \qquad \text{(A-5a)}$$

$$\sigma_a=\sigma_M+\sigma_0\sin(2\pi\nu t) \qquad \text{(cyclic)} \qquad \text{(A-5b)}$$

with ν the cyclic frequency, σ_M and σ_0 the mean and half-amplitude stresses.

The computer algorithm starts with the indentation flaw in its immediate, postindentation state, and grows the crack stepwise in accordance with Eqs. (A-1) to (A-5) until an instability condition $dG_*/dc>0$ is attained (with care to ensure that the program is allowed to continue if this instability leads to a metastable arrest configuration[23]).

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